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(54) IMPROVEMENTS IN THE FINISHING OF TEXTILE MATERIAL

(71) We, HENKEL & CIE. GmbH, a Germany Company, of 67, Henkelstrasse, Duesseldorf, 4000, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a process for finishing textile materials containing wool and especially for the prevention of felting in these materials.

A large number of different compounds has already been proposed for the anti-felting treatment of textiles containing wool. Examples of these include polycondensable compounds such as polyamides, polyurethanes, polycarbamides, polyesters, or polycarbonates, synthetic soft polymers such as polyacrylates, polyethylenes, polyvinyl acetates, polyamide-epchlorhydrin compounds, polyolefines and polyethyleneimines, and isocyanates from polar solvents, such as, for example pyridine or dimethylsulphoxide. Apart from these, further processes are used for the anti-felting treatment for example, the chlorinating or oxidative pretreatment of the wool. All these known processes, however, have the great disadvantage that the textiles so treated obtain a hard and unpleasant handle, so that an additional treatment with restoring agents is required.

The object of the invention is to treat wool-containing textiles in organic solvents so that they may be free from felting and shrinking without special treatment with restoring agents being necessary.

According to the present invention there is provided a process for finishing a textile material which is comprised wholly or substantially of wool, comprising impregnating said textile material with an organic solvent solution of a reaction product containing one free isocyanate group which is obtained by the reaction of a polyisocyanate with a higher molecular weight compound which contains from 8 to 30 carbon atoms, and has at least one reactive hydrogen atom capable of reacting with an isocyanate group, the textile material subsequently being dried.

The polyisocyanates used in the preparation of the reaction product are derived from aliphatic, cycloaliphatic or aromatic isocyanates. Examples of these include hexamethylene diisocyanate, tetramethylene diioscyanate, cyclohexane diisocyanate, hexahydrotolylene diisocyanate, *p*-phenylene diisocyanate and diphenylmethane diisocyanate.

Examples of the higher molecular weight compound which contains 8 to 30 carbon atoms and at least one hydrogen atom which reacts with isocyanate groups, include: higher molecular weight, straight- or branched-chain, saturated or unsaturated, aliphatic alcohols, especially those with 12 to 22 carbon atoms, preferably fatty alcohols of chain lengths C_{12}—C_{20}, prepared from natural fatty acids or fatty acid mixtures, such as for example, are obtained from tallow fats; oleyl alcohol or fatty alcohol mixtures containing oleyl alcohol; alkylphenols containing 8 to 12 carbon atoms in the alkyl chain, for example nonyl phenol; higher molecular weight saturated or unsaturated aliphatic carboxylic acids, especially those with 10 to 22 carbon atoms, preferably fatty acid mixtures of chain lengths C_{12} to C_{20} obtained from natural fats or oils such as are obtained from coconut or tallow fats; oleic acid and fatty acid mixtures containing oleic acid; fatty acid amides and alkanolamides having a chain length of from 12 to 16 carbon atoms and also products of addition of 1 to 4 mol of an alkylene oxide, preferably ethylene oxide or propylene oxide, to the said alcohols, alkylphenols, carboxylic acids or carboxylic acid amides, also alkylmercaptans, for example dodecylmercaptan, aliphatic and aliphatic-aromatic sulphonic acid amides such as dodecylbenzenesulphonamide.

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The process for the production of the reaction product is effected in the presence or absence of organic solvents such as, for example, halogenated hydrocarbons with 1 to 3 carbon atoms, low-boiling esters such as ethyl or butyl acetates, or dimethyl formamide, for 2 to 60 minutes, preferably about 10 minutes, at 20° to 120°C. The relative proportions of the reaction components are chosen so that 0.01 to 0.5, preferably 0.05 to 0.2, reactive hydrogen atoms are present to each isocyanate group.

Liquid or solid reaction products or mixtures soluble in organic solvents are formed which, owing to their content of free isocyanate groups, and possibly unreacted polyisocyanate, are able to react with the wool keratin and in the presence of moisture to produce polymerisation.

The reaction products are applied in an organic solvent. Such solvents are preferably halogenated hydrocarbons with 1 to 3 carbon atoms, such as are used as solvents in chemical cleaning. Examples of such solvents include tetrachlorethylene, trichlorethylene, carbon tetrachloride, 1,2-dichloroethane, methylene chloride, difluorodichloroethane, monofluorotrichloromethane, 1,1,1-trichloroethane, trifluorotrichloroethane and the like. It has been found advantageous to dissolve the reaction products, before dissolving in the said solvents, in a mixture of these solvents which contains in addition at least about 20% by weight of dimethyl formamide. In order to avoid premature setting, 0.01 to 0.1% by weight of phenyl - β - naphthylamine may be added to the solution.

The impregnating of the wool-containing textiles is suitably carried out in the cleaning machines normally used for chemical cleaning, possibly following a chemical cleaning. It is also possible to full the wool before the anti-felting treatment in a solvent bath which contains small amounts of an anionic or non-ionic cleaning intensifier as well as water.

The baths used for the treatment advantageously should contain 1 to 120 preferably 5 to 40 g. per litre of the reaction mixture, through in some instances, it is preferred that the baths contain 1 to 60, especially 3 to 40, or 5 to 120 g. per litre of the reaction product. The treatment is preferably effected at 15° to 35°C. and generally requires a period of 2 to 10 minutes. Following the treatment, the textile may be centrifuged until it contains a residual liquor content of 30 to 60% and may be dried at temperatures from 20° to 80°C. It is advisable to steam the treated textile material after drying in order to remove creases and fold and to give it a good appearance. The textile is then suitably stored for several days, preferably about a week, at room temperature, in order to obtain a final fixing of the condensation products applied.

The process according to the invention leads to a deposition of polymerised isocyanate reaction products on the wool fibres, the passages existing between the edges of the flakes and the shaft of the wool fibre being preferably filled. As a result a marked resistance to felting and creasing is attained by the wool as well as an outstanding and permanent softness and therefore further use of restoring agents is not generally required. Special effects, however, can be attained by the co-use of the usual softeners, synthetic resins, waterproofing agents and the like.

The treatment according to the invention resists normal washing with aqueous washing liquors, as well as treatment in chemical cleaning baths.

A further advantageous result of the treatment is that fixing of the structure of the wool fibre or fabric can be obtained, especially when a heat treatment is applied after the anti-felting treatment. From this it is possible to fix desired alterations in shape permanently in the fabric by pressing or ironing, such as pleating or creasing. These changes in form are also resistant to washing and cleaning.

Example 1

6 kg of woollen knitwear were treated in a cleaning machine of the "Böwe R 9-maxima" ("Böwe" is a Registered Trade Mark type in 60 litres of perchloroethylene with addition of 800 g of a reaction product, which has been prepared from 700 g of hexamethylene-diisocyanate and 100 g of nonylphenol by heating for 30 minutes at 90°C. After centrifuging to a residual moisture content of 60%, the wool was dried at 60°C.

After storing for one week, the treated knitwear was washed in a usual domestic washing machine together with an untreated piece of material for 30 minutes at 60°C with addition of 1.5 per litre of a commercial fine washing agent, centrifuged and dried in the air. As the folowing Table shows, the treated textile has a substantially smaller surface shrinkage.

Material	surface shrinkage in %
untreated goods	14
treated goods	3

The treated goods possessed a pleasant soft handle.

Example 2

In the cleaning plant mentioned in Example 1, 6 kg of woollen knitwear were cleaned as usual in perchloroethylene. After pumping off

the liquid 1320 c.c. of the solution described below were sprayed on the textiles with a spraying device. The solution consisted of 1200 c.c. of perchloroethylene, 30 c.c. of dimethyl formamide, 14 c.c. of *n*-butyl acetate, 16 c.c. of methylene chloride and a reaction product prepared by heating for half an hour at 90°C, 54 g of *p*-phenylene diisocyanate and 6 g of coconut fatty acid monoethanolamide. After the spraying was finished, levelling was effected by allowing the machine to run for 7 minutes, and the material was then dried as usual at 70°C.

The treated knitwear was then pleated, while for comparison a piece of untreated material was also pleated. The pleated articles were then washed as described in Example 1. In contrast to the untreated material the pleats in the treated material were not altered by the washing, and there was just a little felting of the treated material. The handle of the treated textile was soft and pleasant.

Mens trousers consisting of wool/polyester (45/55) were treated in the same way and the creases fixed by pressing. The creases so produced were not altered by a wet wash, as described in Example 1, nor by the usual chemical cleaning.

Example 3

18 kg. of wool pullovers were treated in a standard cleaning machine in 160 litres of perchloroethylene for 5 minutes. The bath contained 2 kg of a reaction product prepared by heating 1.5 kg of 1,4-cyclohexane diisocyanate and 500 g of tallow fatty alcohol of chain lengths C_{16} to C_{18} at 90°C for 40 minutes.

Before applying the treatment bath, the reaction product was first dissolved in 2 kg of a solvent which consisted of two parts of dimethyl formamide, one part of *n*-butyl acetate and one part of methylene chloride.

Following the treatment process, the bath liquor was pumped into the stock tank and the pullovers were centrifuged to a residual moisture content of 40%. After drying at 50°C and storage for 5 days, a pullover together with an untreated pullover was washed five times in the way described in Example 1. It may be seen from the following Table that the alterations in size of the treated pullover are substantially less than those of the untreated pullovers:

Wool pullover	surface Shrinkage in %
untreated, after washing 5 times	29
treated after washing 5 times	7

The treated pullovers possessed an outstanding soft and pleasant handle.

Example 4

15 kg of woollen knitted pullovers were treated for 5 minutes in 130 litres of perchloroethylene in standard cleaning machine. The treatment bath contained 15.6 kg of a reaction product, prepared by heating at 40°C for 20 minutes, 10 kg of hexamethylene diisocyanate and 5.6 kg of a fatty alcohol, prepared from the fatty acids contained in rape oil. The product was dissolved in 35 litres of a solvent which consisted of 2.5 parts of dimethyl formamide and 10 parts of methylene chloride. To increase its stability, 0.03% by weight of phenyl - β - naphthylamine was added to this mixture. After the treatment process was finished, the liquor was pumped back into the stock vessel and the material was centrifuged and dried, as indicated in Example 3.

The woollen pullovers had a soft handle and, compared with the untreated goods, showed remarkably little felting.

WHAT WE CLAIM IS:—

1. A process for finishing a textile material which is comprised wholly or substantially of wool, comprising impregnating said textile materials with an organic solvent solution of a reaction product containing one free isocyanate group which is obtained by the reaction of a polyisocyanate with a higher molecular weight compound, which contains from 8 to 30 carbon atoms, and has at least one reactive hydrogen atom capable of reacting with an isocyanate group, the textile material subsequently being dried.

2. A process as claimed in claim 1 in which the said isocyanate is hexamethylene diisocyanate, 1,4-cyclohexane diisocyanate or *p* - phenylene - diisocyanate.

3. A process as claimed in claim 1 or 2 in which said higher molecular weight compound is a straight or branched chain, saturated or unsaturated, aliphatic alcohol having 12 to 22 carbon atoms.

4. A process as claimed in claim 3 in which the said higher molecular weight compound is a saturated or unsaturated fatty alcohol having a chain length of from C_{12} to C_{20}, or a mixture of such fatty alcohols.

5. A process as claimed in claim 1 or 2 in which said higher molecular weight compound is a fatty acid amide or fatty acid alkanolamide, having a chain length of from 12 to 16 carbon atoms, or mixture thereof, or an alkyl phenol.

6. A proces as claimed in claim 5 in which any alkyl phenol is nonylphenol.

7. A process as claimed in any of claims 1 to 6 in which the reaction between the isocyanate and the higher molecular weight compound is effected at a temperature of from 20° to 120°C.

8. A process as claimed in any of claims 1 to 7 in which the reaction between the iso-

cyanate and the higher molecular weight compound is effected in the presence of an organic solvent.

9. A process as claimed in any of claims 1 to 8 in which the proportion of isocyanate to higher molecular weight compound in their reaction mixture is such that there are 0.01 to 0.5 reactive hydrogen atoms present to each isocyanate group.

10. A process as claimed in claim 9 in which there are 0.05 to 0.2 reactive hydrogen atoms present to each isocyanate group.

11. A process as claimed in any of claims 1 to 10 in which said reaction product is present in said organic solution in a concentration of from 1 to 120 g. per litre.

12. A process as claimed in claim 11 in which said reaction product is present in said solution in a concentration of from 5 to 40 g. per litre.

13. A process as claimed in any of claims 1 to 10 in which said reaction product is present in said organic solution in a concentration of from 1 to 60 g. per litre.

14. A process as claimed in any of claims 1 to 10 in which said reaction product is present in said organic solution in a concentration of from 3 to 40 g. per litre.

15. A process as claimed in any of claims 1 to 14 in which the organic solvent containing said reaction product is a halogenated hydrocarbon having from 1 to 3 carbon atoms.

16. A process as claimed in claim 15, in which said halogenated hydrocarbon is perchlorethylene.

17. A process as claimed in any of claims 1 to 16 in which the reaction product is initially dissolved in a mixture of the organic solvent and dimethyl formamide the latter being present in an amount of at least 20% by weight.

18. A process as claimed in any of claims 1 to 17 in which said solution of the reaction product has a content of phenyl - β - naphthylamine.

19. A process as claimed in claim 18 in which said phenyl - β - naphthylamine is present in an amount of from 0.01 to 0.1% by weight.

20. A process as claimed in any of claims 1 to 19 which is effected in a standard chemical cleaning machine optionally subsequent to a chemical cleaning operation.

21. A process as claimed in claim 1, substantially as hereinbefore described with reference to any of the foregoing Examples.

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